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MEMORANDUM

DATE: 3 December 1998

TO: David Bennett, WAM, U.S. EPA, Region X

FROM: Michelle Turner, Chemist, WESTON, Seattle
Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT: Validation of Organotin Data
Laboratory Batch: K9805481
Site Duwamish River

WORK ASSIGNMENT NO: 46-35-0JZZ

WORK ORDER NO : 4000-019-038-5200-00

DOC. CONTROL NO.: 4000-019-038-AAAK

cc: Bruce Woods, RAP-WAM, U.S. EPA, Region X
Dena Hughes, Site Manager, WESTON, Seattle (memo only)
Kevin Mundell-Jackson, Database Management, WESTON

The quality assurance review of five sediment samples, laboratory batch K9805481, collected from the Duwamish River has been completed. The sediment samples were analyzed for organotins by Columbia Analytical Services of Kelso, Washington. Samples were analyzed by gas chromatography with an FPD detector. The samples were numbered:

98334037	98334043	98334047	98334048
98334056			

Data Qualifications

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract. The review follows the format described in the *National Functional Guidelines for Organic Data Review* (EPA OSWER Directive 9240.1, February 1994), modified to include specific requirements of analytical methods.

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DCN 4000-019-038-AAAK

3 December 1998
Region X





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1. Timeliness

Holding time limits of 7 days for sample extraction and additional 7 days for analysis were established in the project Sampling and Analysis plan. All samples met holding time criteria.

2. Detection Limits - Acceptable

Instrument detection limits met project required quantitation limits

3 Initial Calibration

A six-point initial calibration was performed prior to each analytical batch. The percent relative standard deviation for the initial calibration was within limits of less than 25 percent RSD.

4. Continuing Calibrations

Continuing calibration check was performed after every 10 samples. All target analytes were within required limits for the continuing calibrations with the percent difference for a mid-range standard less than 25 percent.

5. Blanks

a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met. No target analytes were reported in laboratory method blanks.

b) Field Blanks

No field blanks were associated with this SDG.

6 Surrogate Compound Recovery

Surrogate recovery goals for tri-n-propyltin were established in the project Sampling and Analysis Plan at 60 to 130 percent for both sediment and porewater. Based on conversations with the laboratory an additional surrogate, tri-pentyltin was added and

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historical laboratory control chart limits were also used for data qualification. Laboratory limits are presented below:

Surrogate Compound	Sediment Limits	Porewater Limits
Tripropyltin	20 - 195%	20 - 113%
Triphenyltin	20 - 172%	20 - 133%

Surrogate compound percent recovery met quality control criteria for all samples, with the exception of the following:

Sample	Surrogate	Percent Recovery
K980818-LCS (8/22)	Triphenyltin	59
K980818-LCS (9/21)	Triphenyltin	50
k980818-MB (9/21)	Tripropyltin	51

Sample results were qualified as estimated (J) when both surrogate recoveries were outside project limits. Surrogate recoveries for samples associated with the method blank and LCS were within QC limits.

7 Laboratory Control Sample (LCS)

LCS recovery goals for butyltins were established in the project Sampling and Analysis Plan at 60 to 130% for both sediment and porewater. Based on conversations with the laboratory, historical control chart limits of 20 to 138 percent for water and 20 to 164 percent for sediment were also used for data qualification.

Laboratory control sample percent recoveries met QC guidelines (P-project, L-laboratory), with the exception of the following.

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LCS	Analyte	Percent Recovery	QC Limit	Associated Samples
K980818-LCS (8/21)	n-Butyltin	20	60-130 (P) 20-164 (L)	98334037 98334043 98334047 98334048 98334056
K980818-LCS (9/21)	n-Butyltin	20	60-130 (P) 20-164 (L)	none

Sample results were qualified as estimated (J) when LCS recoveries were outside project limits. Undetected results were qualified as estimated (UJ) when LCS recoveries were outside project limits

8. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

The following matrix spike recovery goals were established in the project Sampling and Analysis Plan at for both sediment and porewater.

Analyte	% Recovery
Tributyltin	40 - 120%
Diethyltin	30 - 120%
Monobutyltin	10 - 129%

All MS/MSD sample percent recoveries and relative percent differences (RPDs) met QC guidelines, with the exception of the following:

Analyte	MS %R	MSD %R	RPD	Associated Samples
K9805457-002MS (Batch QC)	155	91	30	98334037 98334043 98334047 98334048 98334056



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MS/MSD recoveries and RPDs were not calculated for Tri-n-butyltin and Di-n-butyltin as the analyte concentration was higher than the spike level. No qualifiers were assigned solely on MS/MSD results.

9. Field Duplicate Analysis

No field duplicates were associated with this SDG.

10. Sample Analysis

A cursory review of raw data was performed. All results were verified on a second, dissimilar, confirmation GC column. A batch QC duplicate was also analyzed with this SDG. The RPD result for Di-n-butyltin was 107 percent. The case narrative noted that the MS/MSD recovery for Tri-n-butyltin and Di-n-butyltin for the batch QC sample was not calculated due to the analyte concentration in the native sample. The narrative also notes that the RPD for the batch QC duplicate was high for Di-n-Butyltin. The batch QC duplicate samples, along with the method blank and LCS, were rederivatized and produced the same results. No other problems were noted.

11. Laboratory Contact

No laboratory contact was required

Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values.

Data Qualifiers

- U - The compound was analyzed for, but was not detected.
- UJ - The compound was analyzed for, but was not detected. The associated quantitation limit is an estimate because quality control criteria were not met
- J - The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because

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concentrations reported are less than the quantitation limit or lowest calibration standard

- R - Quality control indicates that data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification.
- N - Presumptive evidence of presence of material (tentative identification)
- I - Elevated reporting limit due to matrix interference.

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805481
Date Collected: 8/13/98
Date Received: 8/14/98

Butyltins

Sample Name 98334037 Units ug/Kg (ppb)
Lab Code K9805481-001 Basis Dry
Test Notes D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	ND	
Tri-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	69	
Di-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	25	
n-Butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	17	J

D

The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By



Date

9/22/98

1S22/020597p

COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805481
Date Collected: 8/13/98
Date Received: 8/14/98

Butyltins

Sample Name 98334043 **Units** ug/Kg (ppb)
Lab Code K9805481-007 **Basis** Dry
Test Notes D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	ND	
Tri-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	28	
Di-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	ND	
n-Butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	6 J	

D

The MRL is elevated because of matrix interferences and because the sample required diluting

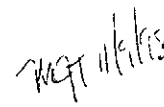
Approved By



Date

9/22/08

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805481
Date Collected: 8/13/98
Date Received: 8/14/98

Butyltins

Sample Name	98334047	Units	ug/Kg (ppb)
Lab Code	K9805481-011	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	ND	
Tri-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	49	
Di-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	ND	
n-Butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	19	J

D The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By TJ Date 9/22/98

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WGT 11/6/98

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805481
Date Collected: 8/13/98
Date Received: 8/14/98

Butyltins

Sample Name 98334048 **Units** ug/Kg (ppb)
Lab Code K9805481-012 **Basis** Dry
Test Notes D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	ND	
Tri-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	120	
Di-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	31	
n-Butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	22	J

D

The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By



Date

9/22/98

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9805481
Date Collected: 8/13/98
Date Received: 8/14/98

Butyltins

Sample Name 98334056 **Units** ug/Kg (ppb)
Lab Code K9805481-020 **Basis** Dry
Test Notes D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	ND	
Tri-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	31	
Di-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	10	
n-Butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	9 J	

D

The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By

Date

9/22/98

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